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## (54) METHOD FOR PRODUCING POLYVINYL ACETAL RESIN

### (57)Abstract:

**PROBLEM TO BE SOLVED:** To provide a method for producing an excellent polyvinyl acetal resin having low content of impure electrolytes by which an incorporation of a neutralized salt or the like generated in the process of neutralizing an acid catalyst in the polyvinyl acetal resin slurry generated by condensation reaction of polyvinyl alcohol with aldehydes in the presence of the acid catalyst in an aqueous phase into particles thereof can be suppressed.

**SOLUTION:** The method for producing the polyvinyl acetal resin is the one of neutralizing and washing the polyvinyl acetal resin product obtained by carrying out the condensation reaction of the polyvinyl alcohol with the aldehydes in the presence of the acid catalyst in the aqueous phase and the steps of the neutralization and washing is carried out repeatedly in plural times. The first time of the neutralizing reaction is simultaneously carried out with the time when the temperature of the obtained polyvinyl acetal resin product reaches glass-transition temperature.

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DETAILED DESCRIPTION

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[Detailed Description of the Invention]

[0001]

[Field of the Invention] This invention relates to the manufacture approach of polyvinyl-acetal resin.

[0002]

[Description of the Prior Art] The polyvinyl-acetal resin represented by polyvinyl butyral resin is used widely in extensive fields, such as a coating, adhesives, and an interlayer for safety glass, with the outstanding adhesive property. Although these polyvinyl-acetal resin carries out the condensation reaction of polyvinyl alcohol and the aldehydes and is obtained An organic solvent is made to distribute polyvinyl alcohol as the manufacture approach. After depositing polyvinyl-acetal resin from the polyvinyl-acetal resin solution which add an aldehyde compound, it was made to react using an acid catalyst, and was generated, The settling which a reaction advances in the aqueous phase, the generated polyvinyl-acetal resin loses solubility, precipitates in the shape of a particle to a product, and is separated into the bottom of existence of the solution process which carries out rinsing desiccation, and an acid catalyst from aqueous intermediation of the system of reaction is known. Since powder-like polyvinyl-acetal resin can be obtained from a water solution at a single process, as compared with the solution process which needs for an excess the process which deposits polyvinyl-acetal resin from the organic solvent solution of the polyvinyl-acetal resin of a product, the above-mentioned settling is easy a process few, and is evaluated as an approach of showing higher productivity.

[0003] However, the acid catalyst used in the particle of the polyvinyl-acetal resin which reaction temperature became high, and the particle of the polyvinyl-acetal resin of a product tended to make settling big and rough, consequently was made big and rough will be incorporated so much.

[0004] When the application of the binder of ceramic system electronic parts etc. is presented in polyvinyl-acetal resin, in order that the quality may be made into a problem and contamination of residue, such as the above-mentioned acid catalyst, may avoid the debasement of the above-mentioned polyvinyl-acetal resin especially, it carries out comparatively the condensation reaction described above by whenever [ low-temperature ], and is atomizing the polyvinyl-acetal resin of a product.

[0005] However, however it may atomize the polyvinyl-acetal resin of a product, it will be difficult to separate the polyvinyl-acetal resin which does not include contamination of the above-mentioned acid catalyst etc. only by rinsing from the obtained slurry-like product, and a neutralization process will usually be included in a rinsing process. That is, the ordinary temperature neutralization which throws in a neutralizer for the acid catalyst in the mother liquor of the slurry-like product obtained by the condensation reaction described above, or after rinsing and reducing acid-catalyst concentration, the above-mentioned neutralizer is thrown in, a neutralization salt and a residual neutralizer are rinsed, it dries and particle-like polyvinyl-acetal resin is produced commercially.

[0006] the appropriate particle-like polyvinyl-acetal resin from which it is alike and neutralization of the above-mentioned acid catalyst was obtained by heat of reaction -- the neutralization more than the glass transition temperature (it calls T<sub>g</sub> for short hereafter) -- constant temperature -- it becomes the temperature of a region and it becomes difficult to carry out the washing removal of the neutralization

salt which the fusion of particles occurred, and a part of neutralization salt and residual neutralizer will be incorporated in a fusion particle, and was incorporated in these fusion particles in the rinsing processing subsequently performed completely. neutralization of the above-mentioned neutralization -- constant temperature -- if the temperature of a region will be reduced, when a neutralizer input will be adjusted, an acid catalyst will remain and the quality problem of polyvinyl-acetal resin will be caused independently.

[0007]

[Problem(s) to be Solved by the Invention] The place which this invention is made in view of the above-mentioned fact, and is made into the purpose controls the incorporation into polyvinyl-acetal resin particles, such as a neutralization salt generated in the process which neutralizes the acid catalyst in the polyvinyl-acetal resin slurry generated by the condensation reaction described above, and is to offer the approach of manufacturing outstanding polyvinyl-acetal resin with few contents of a contamination electrolyte.

[0008]

[Means for Solving the Problem] The manufacture approach of the polyvinyl-acetal resin invention according to claim 1 is the manufacture approach of the polyvinyl-acetal resin which neutralizes the polyvinyl-acetal resin product which was made to carry out the condensation reaction of polyvinyl alcohol and the aldehydes in the aqueous phase to the bottom of existence of an acid catalyst, and was obtained, and is rinsed, and the above-mentioned neutralization and a rinsing process are performed repeatedly two or more times, and it carries out at the same time the polyvinyl-acetal resin product from which first-time neutralization was obtained reaches the glass transition temperature of this resin product.

[0009] The manufacture approach of the polyvinyl-acetal resin invention according to claim 2 is the manufacture approach of the polyvinyl-acetal resin invention according to claim 1, and after the neutralization performed after first-time neutralization rinsing reaches the temperature more than the glass transition temperature of the obtained polyvinyl-acetal resin, it is carried out.

[0010] The polyvinyl alcohol used by the manufacture approach of the polyvinyl-acetal resin of this invention and aldehydes are acetalized by the condensation reaction in the aqueous phase under existence of an acid catalyst, and it will not be limited especially if polyvinyl-acetal resin is obtained from these. The above-mentioned acid catalyst will not be limited especially if the catalyst of the above-mentioned acetalization reaction is carried out good.

[0011] Although the above-mentioned neutralization which is in the manufacture approach of the polyvinyl-acetal resin of this invention, and is performed repeatedly two or more times, and a rinsing process point out that neutralization processing is performed and the process by which rinsing processing is carried out is performed twice [ at least ] or more Even when rinsing processing covers multiple times to one neutralization processing so that the above-mentioned neutralizer may be thrown in and rinsing processing may be carried out further after rinsing first and reducing acid-catalyst concentration, the above-mentioned neutralization and a rinsing process are counted among 1 time from the contents of neutralization processing.

[0012] The manufacture approach of the polyvinyl-acetal resin invention according to claim 1 The polyvinyl-acetal resin product which was made to carry out the condensation reaction of polyvinyl alcohol and the aldehydes in the aqueous phase to the bottom of existence of an acid catalyst, and was obtained is neutralized. It is the manufacture approach of the polyvinyl-acetal resin to rinse. The above-mentioned neutralization and a rinsing process It is carried out repeatedly two or more times. First-time neutralization The neutralization salt and residual neutralizer which were generated before the fusion of the particles of polyvinyl-acetal resin was prevented or fusion occurred, since the obtained polyvinyl-acetal resin product is carried out at the same time it reaches Tg of this resin product are washing style SARERU. Therefore, the neutralization salt and residual neutralizer which adhered to the particle of polyvinyl-acetal resin, or were incorporated in this particle serve as a minute amount extremely, according to the neutralization performed repeatedly and a rinsing process, the amounts of contamination, such as a neutralization salt and a residual neutralizer, serve as a low more, and the

engine performance high as a binder of the ceramics with which electronic parts, such as a semiconductor device, etc. are presented etc. can be demonstrated.

[0013] The manufacture approach of the polyvinyl-acetal resin invention according to claim 2 Since it is carried out after the neutralization which is the manufacture approach of the polyvinyl-acetal resin invention according to claim 1, and is performed after first-time neutralization rinsing reaches the temperature more than Tg of the obtained polyvinyl-acetal resin the acid catalyst which is almost removed and remains slightly by first-time neutralization rinsing -- neutralization -- constant temperature -- since neutralization processing is carried out with a new neutralizer in the temperature more than Tg of the polyvinyl-acetal resin at the time, it is neutralized almost completely and removed from polyvinyl-acetal resin by rinsing.

[0014] the above-mentioned neutralization processing -- setting -- polyvinyl-acetal resin -- the neutralization more than the Tg -- constant temperature -- generating of the fusion of these resin particles, although it is since it is put to the temperature of a region Since it is after the neutralization already performed repeatedly and a rinsing process, impurity, such as a neutralization salt incorporated in part in the fusion particle, also serves as ultralow volume, and the residual neutralization salt and the amount of residual neutralizers in the polyvinyl-acetal resin particle after rinsing and desiccation are ultralow volume.

[0015]

[Embodiment of the Invention] Hereafter, although the example of this invention is explained, this invention is not limited only to these examples.

[0016] (Example 1) With the conventional method, polyvinyl alcohol 283g of 97.5% of saponification degrees and a degree of polymerization 360 was added to 2657g of pure water, and was stirred and dissolved in it. 364g of hydrochloric acids of 35 % of the weight of concentration and n-butylaldehyde 196g were added to the obtained polyvinyl alcohol water solution, the acetalization reaction was performed, and the polyvinyl-acetal resin slurry which consists of a white particle was obtained. The appearance particle diameter of the obtained polyvinyl-acetal resin particle was [ 74 mol % and Tg of 10-20 micrometers and the degree of acetalization ] 61 degrees C.

[0017] After having rinsed until the chlorine content in the obtained polyvinyl-acetal resin slurry mother liquor was set to 10 ppm, and reducing acid-catalyst concentration, the polyvinyl-acetal resin slurry 4000 weight section was adjusted so that this polyvinyl-acetal resin slurry concentration might become 10% of the weight, and it heated until the temperature of a neutralization processor became 61 degrees C, adding and stirring the soda ash 4 weight section. When the temperature of the above-mentioned neutralization processor amounted to 61 degrees C, the neutralization salt, residual neutralizer, etc. which generated by performing rinsing processing immediately were removed. The first neutralization and the chlorine content in the polyvinyl-acetal resin slurry mother liquor after rinsing were 10 ppm, and the sodium content was 6 ppm.

[0018] subsequently -- until it adjusts the polyvinyl-acetal resin slurry 4000 weight section in which the first neutralization and rinsing processing were performed so that this polyvinyl-acetal resin slurry concentration may become 10% of the weight, it adds the soda ash 0.5 weight section, and the temperature of a neutralization processor becomes 65 degrees C, stirring -- heating -- neutralization -- after taking constant temperature for 3 hours, the neutralization salt, residual neutralizer, etc. which generated by performing rinsing processing were removed. The second neutralization and the chlorine content in the polyvinyl-acetal resin slurry mother liquor after rinsing were 10 ppm, and the sodium content was 8 ppm. The second neutralization and the polyvinyl-acetal resin slurry by which rinsing processing was carried out were dried, and white particle-like polyvinyl-acetal resin was produced. 80-100 micrometers and a chlorine content were [ 59 ppm and the sodium content of the appearance particle diameter of the obtained polyvinyl-acetal resin ] 46 ppm.

[0019] (Example 1 of a comparison) the temperature of the first neutralization of an example 1, and the neutralization processor of rinsing processing -- 61 degrees C to 65 degrees C -- changing -- neutralization -- except having performed rinsing processing, like an example 1, perform neutralization and rinsing processing and pass a desiccation process after one neutralization and rinsing processing,

after taking constant temperature for 3 hours -- white particle-like polyvinyl-acetal resin was produced. The chlorine content in the polyvinyl-acetal resin slurry mother liquor after neutralization and rinsing processing was 8 ppm, and the sodium content was 7 ppm. Moreover, 80-100 micrometers and a chlorine content were [ 320 ppm and the sodium content of the appearance particle diameter of the obtained polyvinyl-acetal resin ] 300 ppm.

[0020] (Example 2) With the conventional method, polyvinyl alcohol 322g of 94.8% of saponification degrees and a degree of polymerization 820 was added to 2597g of pure water, and was stirred and dissolved in it. 364g of hydrochloric acids of 35 % of the weight of concentration and n-butyraldehyde 217g were added to the obtained polyvinyl alcohol water solution, the acetalization reaction was performed, and the polyvinyl-acetal resin slurry which consists of a white particle was obtained. The appearance particle diameter of the obtained polyvinyl-acetal resin particle was [ 74 mol % and Tg of 10-20 micrometers and the degree of acetalization ] 60 degrees C.

[0021] Subsequent processes produced white particle-like polyvinyl-acetal resin through the desiccation process in the second neutralization and a rinsing process list like the example 1 except having changed whenever [ stoving temperature / at the time of the first neutralization and the neutralization in a rinsing process ] into 60 degrees C. The first neutralization and the chlorine content in the polyvinyl-acetal resin slurry mother liquor after rinsing were 8 ppm, the sodium content was 6 ppm, the second neutralization and the chlorine content in the polyvinyl-acetal resin slurry mother liquor after rinsing were 9 ppm, and the sodium content was 8 ppm. Moreover, 80-100 micrometers and a chlorine content were [ 120 ppm and the sodium content of the appearance particle diameter of the obtained polyvinyl-acetal resin ] 96 ppm.

[0022] (Example 2 of a comparison) the temperature of the first neutralization of an example 2, and the neutralization processor of rinsing processing -- 60 degrees C to 65 degrees C -- changing -- neutralization -- except having performed rinsing processing, like an example 1, perform neutralization and rinsing processing and pass a desiccation process after one neutralization and rinsing processing, after taking constant temperature for 3 hours -- white particle-like polyvinyl-acetal resin was produced. The chlorine content in the polyvinyl-acetal resin slurry mother liquor after neutralization and rinsing processing was 10 ppm, and the sodium content was 8 ppm. Moreover, 80-100 micrometers and a chlorine content were [ 420 ppm and the sodium content of the appearance particle diameter of the obtained polyvinyl-acetal resin ] 390 ppm.

[0023]

[Effect of the Invention] The manufacture approach of the polyvinyl-acetal resin invention according to claim 1 In neutralization down stream processing of the polyvinyl-acetal resin slurry which was made to carry out the condensation reaction of polyvinyl alcohol and the aldehydes in the aqueous phase to the bottom of existence of an acid catalyst, and was obtained since it was constituted as mentioned above. Before the fusion of the particles of polyvinyl-acetal resin is prevented or fusion occurs The neutralization salt and residual neutralizer which adhered to the particle of polyvinyl-acetal resin since it was what flushes the neutralization salt and residual neutralizer which were generated, or were incorporated in this particle serve as a minute amount extremely, and according to the neutralization performed repeatedly and a rinsing process The amounts of contamination, such as a neutralization salt and a residual neutralizer, serve as a low more, and the engine performance high as a binder of the ceramics with which electronic parts, such as a semiconductor device, etc. are presented etc. can be demonstrated.

[0024] the acid catalyst which is almost removed and remains slightly by previous neutralization rinsing while doing so the effectiveness described above, since the manufacture approach of the polyvinyl-acetal resin invention according to claim 2 is constituted as mentioned above -- neutralization -- constant temperature -- since neutralization processing is carried out with a new neutralizer in the temperature more than Tg of the polyvinyl-acetal resin at the time, it is neutralized almost completely and removed from polyvinyl-acetal resin by rinsing.

[0025] the above-mentioned neutralization processing -- setting -- polyvinyl-acetal resin -- the neutralization more than the Tg -- constant temperature -- since it is put to the temperature of a region,

there is generating of the fusion of these resin particles, but since it is after the neutralization already performed repeatedly and a rinsing process, impurity, such as the neutralization salt incorporated in part in a fusion particle, also serves as ultralow volume, and a residual neutralization salt and the amount of residual neutralizers have become with ultralow volume in the polyvinyl-acetal resin particle after rinsing and desiccation.

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CLAIMS

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[Claim(s)]

[Claim 1] It is the manufacture approach of polyvinyl-acetal resin that it is the manufacture approach of the polyvinyl-acetal resin which neutralizes the polyvinyl-acetal resin product which was made to carry out the condensation reaction of polyvinyl alcohol and the aldehydes in the aqueous phase to the bottom of existence of an acid catalyst, and was obtained, and is rinsed, the above-mentioned neutralization and a rinsing process are performed repeatedly two or more times, and it is characterized by to carry out at the same time the polyvinyl-acetal resin product from which first-time neutralization was obtained reaches the glass transition temperature of this resin product.

[Claim 2] The manufacture approach of the polyvinyl-acetal resin according to claim 1 characterized by carrying out after the neutralization performed after first-time neutralization rinsing reaches the temperature more than the glass transition temperature of the obtained polyvinyl-acetal resin.

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[Translation done.]